Advanced Casting Research Center (ACRC) Consortium Meeting

May 15, 2001

Report 01-#1

Metal Processing Institute
WPI, Worcester, MA 01609 USA
www.wpi.edu/+mpi

TABLE OF CONTENTS

A. Microstructural Evolution in Semi-Solid Alloys (MIT)

MICROSTRUCTURAL EVOLUTION IN SEMI-SOLID ALLOYS

Report PR-01-#1

Research Team:	Merton C. Flemings	617-253-3233	flemings@mit.edu
	James Yurko	617-258-5547	yurko@mit.edu
	Raul Martinez	617-258-5547	raulm@mit.edu
Focus Group:	Craig Bergsma	(Northwest Aluminum)	
	Stig Brusethaug	(Hydro Aluminum A.S.)	
	Alain Charbonnier	(Aluminum Pechiney)	
	Karuppa Chinnathamba	(Citation Aluminum)	
	Tom DeJohn	(Superior Industries International)	
	Michel Garat	(Aluminum Pechiney)	
	Vincent Genise	(Citation Aluminum)	
	John Jorstad	(JLJ Technologies)	
	John Miller		
	Rod Riek	(Harley-Davidson Motor)	
	Jim Van Wert	(Amcast)	

PROJECT STATEMENT

Objectives

- Obtain fundamental rheological data on microstructural evolution

- Design experiments to obtain information of direct applicability to both the modeling studies at WPI and to the planned experiments for industrial type forming applications

- Determine the effects of semi-solid "slurry" structure on flow at the high shear rates representative of actual forming processes.

- Develop new methods of forming structures for SSF

Strategy

- Current descriptions of the flow behavior of semi-solid materials are for highly idealized conditions, which are not encountered in industrial forming processes. This project examines the high shear rate, transient flow behavior of both rheocast and thixocast alloys.

- Two complementary approaches are being employed to examine the flow behavior of semi-solid metals under experimental conditions that are closer to those found in industrial practice. In the first approach, a "drop forge" viscometer is being employed to examine the flow behavior under very rapid compression rates of A357, A356 diluted with pure Al, and Al-4.5Cu alloys. The A357 alloys under investigation are of commercial origin (MHD and SIMA processes) and the rheocast modified A356 and Al-4.5Cu alloys are produced by a process developed at MIT. In the second approach, a modified parallel-plate rheometer was used to measure viscosities of commercial A357 slurries. The actual slurries were produced by rapid reheating of MHD material in the rheometer. The combined approaches allow transient rheological measurements to be carried out over a broad range of solid content and under conditions closer to those of actual forming processes.

- In an added activity, the mechanism of formation of structures suitable for semi-solid forming is being investigated, with the aim of determining improved methods of producing these structures.

ACHIEVEMENTS THIS QUARTER

Drop Forge Viscometer
Viscosity calculations of rheocast, modified A356 was conducted up to volume fraction solid of 0.67. A357 MHD material was examined for comparison with SIMA material. Slow compression studies were carried out with the DFV on A357 SIMA, and segregation was observed in the compressed samples.

Parallel-Plate Rheometer
Work completed in January 2001

Formation of SSM Structures
Non-dendritic A356 Aluminum alloy was produced by a new approach (New MIT Process): processing with rapid cooling and vigorous agitation during only the first few degrees of solidification. Experiments were conducted to test how much solid must form during processing to create the non-dendritic structure. Other experiments tested the required intensity of liquid flow to achieve these structures.

CHANGES IN PROJECT STATEMENTS

None

WORK PLANNED FOR NEXT QUARTER

Drop Forge Viscometer

The segregation phenomenon will be examined, along with the continuation of modeling work to verify existing flow models.

Structure Formation (New MIT Process)

Design and build highly instrumented equipment to further characterize the solidification conditions that lead to non-dendritic structure. Begin heat and fluid flow modeling to aide experimental work in determining required solidification conditions.

OPERATIONAL SCHEDULE

	June 2001	July 2001	Aug. 2001	Sept. 2001
DROP FORGE VISCOMETER				
Experimentally examine segregation phenomenon				
Compete modeling work and wrap up study				
FORMATION OF SSM STRUCTURES				
Design and build highly instrumented equipment for further tests				
Model heat and fluid flow to understand required conditions				

MICROSTRUCTURAL EVOLUTION IN SEMI-SOLID ALLOYS

M. C. Flemings, J. Yurko, and R. Martinez
Dept. of Materials Science and Engineering
Massachusetts Institute of Technology

Summary

James Yurko is completing his doctoral thesis this month, on rapid transient measurement of rheology of semi-solid alloys, using his newly developed Drop Forge Viscometer ("DFV"). Raoul Martinez is completing his master's degree on his newly developed approach to producing semi-solid alloys of the desired non-dendritic structure (the "New MIT Process"). The following summary report of MIT activities comprises the Abstract and Conclusions of those two theses.

Rapid Transient Measurement of Rheological Behavior of Semi Solid Aluminum Alloys (J. Yurko)

Abstract

The rheological behavior and microstructure of semi-solid aluminum alloys were studied using a novel apparatus, the Drop Forge Viscometer (DFV). The viscometer determines force from the curvature of displacement data allowing calculations of viscosities at shear rates in excess of 10^3 s^{-1}. Alternatively, the DFV can be operated like a conventional parallel-plate compression viscometer, attaining shear rates as low as 10^{-5} s^{-1}. Durations of an experiment range between approximately 5 ms and 24 hours.

Most rapid compression tests had periods of first rapidly increasing shear rate followed by rapidly decreasing shear rate. Viscosity during the increasing shear rate period decreased by 1-2 orders of magnitude. The viscosity during the decreasing shear rate was an order of magnitude larger than another that had achieved a 75% greater maximum shear rate.

The DFV was used to calculate viscosity as a function of shear rate for Al-Si and Al-Cu alloys that were rheocast with the commercial SIMA and MHD processes, as well as the recently developed MIT method. Experiments were conducted between fractions solid of 0.44 and 0.67. Viscosity of A357 produced by the three processing routes all had similar viscosities, ranging from 300 Pa.s at 120 s^{-1} to 2.2 Pa.s at 1500 s^{-1}. The final height of compressed Al-Cu was always greater than Al-Si for a given set of experimental conditions.

Segregation was not observed in rapid compression experiments of less than 10 ms, either visually or with EDS characterization. At low compression velocities, segregation was observed and increased with the amount of strain.

Conclusions

1. A novel apparatus, the Drop Forge Viscometer (DFV) was designed and constructed based on the parallel-plate compression viscometer. The viscometer permits calculating force from the second derivative of the displacement data allowing calculations of viscosities at shear rates in excess of 10_ s^{-1}. Total duration of a test can be less than 10 ms. Alternatively, the DFV can be operated as a conventional parallel-plate viscometer, attaining shear rates as low as 10^{-5} s^{-1}.

2. A unique feature of the DFV is that a typical experiment yields instantaneous, volume-averaged viscosity first under rapidly increasing shear rate and then under rapidly decreasing shear rate.

3. The apparatus was used to determine the viscosity as a function of shear rate and fraction solid for alloys of non-dendritic structure produced by three means, known as the SIMA, MHD, and MIT processes. The viscosity of the MIT material was similar to alloys produced by the commercial SIMA and MHD processes.

4. Most compression tests in this work (all of the rapid compression tests) were completed within about 6 ms. During the increased shear rate portion of a typical test (lasting about 4 ms), the viscosity dropped by 1-2 orders of magnitude. Viscosity during the decreasing shear rate period decreased with increasing maximum increasing shear rate. For example, in two SIMA alloys of about 0.48 volume fraction solid (g_s), the viscosity calculated at 100 s^{-1} was an order of magnitude greater than a sample that achieved a 75% higher shear rate.

5. Viscosity versus increasing shear rate of the Al-Si alloys at 0.48 g_s produced by the three different process routes all showed roughly the same viscosity as a function of shear rate, ranging from 300 Pa.s at 120 s^{-1} to 2.2 Pas at 1500 s^{-1}.

6. Viscosity as a function of increasing shear rate of the Al-4.5wt%Cu alloy was larger than an Al-Si alloy for a fraction solid of about 0.48, but the viscosity was about the same as an Al-Si alloy at 0.56 g_s. The final compressed height of the Al-Cu was always greater than an Al-Si alloy for similar experimental conditions.

7. Visual examination of cross sections did not show separation of liquid and solid phases in any of the rapidly compressed samples in the shear rate range of 10 to 1500 s^{-1}. Segregation was not detected by either quantitative

metallography or chemical composition variations in an A357 SIMA sample that had been compressed at a fraction solid of 0.48 and at shear rates ranging between 15 and 750 s^{-1}. However, when the DFV was used as a conventional parallel-plate viscometer to achieve very low shear rates, there was a fraction solid difference from center to edge of 0.20 (calculated from variation in chemical composition) after compression for 15 minutes in the shear rate range of 10^{-2} to 10^{-4} s^{-1}. A sample that was compressed for four hours at shear rates of 10^{-2} to 10^{-5} s^{-1} had a fraction solid of approximately zero at the edge and 0.70 in the center. Segregation appears to increase with increasing strain in the low shear rate experiments

8. The maximum fraction solid that was compressed with the DFV at high and low compression velocity was 0.67 and 0.70, respectively.

9. The DFV is a useful process control tool for comparing the relative compression behavior of semi-solid alloys in the two-phase temperature region. Differences between samples are reflected in the final shape and thickness of the compressed samples, and also in the displacement data.

The DFV provides unique transient rheological data of particular relevance to the modeling of semi-solid processing. Validation of rheological models was achieved by comparing experimental results with model predictions

SSM Structure Formation (Raoul Martinez)

Abstract

A crucial aspect for the commercial use of semisolid forming technology is the economical production of alloys with non-dendritic microstructure. There are currently available a variety of processing routes to obtain these special structures, but each has disadvantages. Attempts are being made to find other simple and effective methods to create non-dendritic material to be used in semisolid forming.

It is now known that the combination of cooling with vigorous agitation during the solidification of an alloy serves to spheroidize the primary solid particles. Several processes apply this combination to create non-dendritic microstructure. What has been unknown up to this point is how much cooling and agitation is required. It is also largely unknown during what portion of the solidification range does processing induce structural changes. The answer to these questions may be critical to the recognition of new processing methods. In this work it is suggested that processing during the very beginning of solidification is most significant for creating non-dendritic structures. A new approach was developed to test this hypothesis.

A rotating copper rod was immersed in a molten aluminum alloy, held just above the liquidus temperature. The rod quickly lowered the temperature of the melt just below the liquidus temperature, and simultaneously stirred the alloy. By removing the rod when the melt temperature had dropped just a few degrees below the liquidus, the combination of stirring and rapid cooling was applied when only small amounts of solid had formed. Results show that good non-dendritic structures can be created by processing in this manner when as little as 1 vol% solid had solidified. No significant microstructural differences were observed when the alloy was stirred by the rod beyond this point in the solidification range. It was determined that all of the rotational speeds used in the experiments induced turbulent liquid flow. The degree of turbulence was not found to significantly affect the microstructures produced.

The following is a brief summary of the conclusions that have been drawn from this work. It has been well documented that providing rapid cooling and vigorous agitation during the initial stages of alloy solidification can create non-dendritic structures. This processing route is a powerful new way to efficiently create semisolid material free of entrapped eutectic. Processing during the solidification of the first 1 vol% solid is most critical in the development of the non-dendritic structure. When the fluid flow during processing is turbulent, the solidified alloy has a non-dendritic structure. Over the range investigated, the rotational speed of the rod was not found to have a significant effect on the sphericity of the primary particles.

Conclusions

A. Process Development

1. A new processing approach for the formation of non-dendritic structures has been developed. The technique involves immersing a rotating copper rod, initially at room temperature, into an A356 melt held just above its liquidus temperature. The rod rapidly cools the alloy below its liquidus to initiate solidification while vigorously stirring the melt. The rod remains in the melt for short periods, just long enough to cool the melt a few degrees below the liquidus temperature.

1. The processing technique used can create semisolid slurries directly from the liquid alloy in a simple and efficient manner.

1. The non-dendritic structures created by this technique have demonstrated a semisolid flow behavior comparable to that of commercially available SIMA material.

1. The material produced by this method is essentially free of entrapped eutectic. This may be a significant advantage when using the material in semisolid forming operations.

2. Although most of the material produced by the new processing technique is non-dendritic, some regions of structural inhomogeneity are present. The formation of these regions is likely caused by stirring with the rod geometry.

B. Required Degree of Solidification During Processing

1. During the onset of solidification, or shortly thereafter, processing can lead to the formation of the non-dendritic structures. These structures have been created when the cooling and agitation induced by the spinning copper rod was applied for a very small solidification interval, such that as little as 1 vol% solid was formed before the rod was removed. No major microstructural differences were observed when the rod was removed after more solid fraction had formed, indicating that processing during the solidification of the first 1 vol% solid is the most critical for creating non-dendritic structure.

2. The previous conclusion was verified by calculating the average shape factor for reheated material which had been stirred for 2, 5, 11, and 20 seconds. These stirring times cooled the melt different amount below the liquidus temperature, corresponding to varying degrees of solidification ranging from 1 to 7 vol% solid. For each stirring time, the particles have a constant shape factor value near 0.75. Since this value is independent of stirring time and the associated degree of solidification, the results support the previous conclusion that microstructural changes must occur by processing during the solidification of the first 1 vol% solid.

C. Required Amount of Liquid Flow During Processing

1. Using the new processing technique, "low" rotational speeds are effective in creating the non-dendritic microstructure. When the copper rod was rotated at only 60 RPM and immersed for 20 seconds, non-dendritic material was produced.

2. After reheating, the average particle shape factor was 0.71 for material stirred with 60 RPM ; only slightly less than the shape factor for material processed with higher rotational speeds of 508 and 1001 RPM which was 0.73 and 0.78, respectively. Reheated dendritic material had an average shape factor of 0.51. The transition between non-dendritic and dendritic morphology must occur when rotational speeds less than 60 RPM are used.

3. By calculating the Taylor number, it was shown that the liquid flow induced by the stirring rod is always in the turbulent flow regime, even when the lowest stirring speed of 60 RPM was used.

B. Development of Alternate Semi-Solid Aluminum Alloys (ORNL)

DEVELOPMENT OF ALTERNATE SEMI-SOLID ALUMINUM ALLOYS

Report PR-01-#1

Research Team:	Srinath Viswanathan	(865) 576-9917,	viswanathans@ornl.gov
	Qingyou Han	(865) 574-4352,	hanq@ornl.gov
	Jason Astle	(508) 831 5992,	clyde@wpi.edu
	Qingyue Pan	(508) 831 5790	qypan@wpi.edu
	Diran Apelian	(508) 831 5992	dapelian@wpi.edu
Focus Group:	Tom Caldwell	(Madson-Kipp)	
	Babu DasGupta	(SPX)	
	Scott Kangas	(Buhler)	
	Tom Prucha	(Intermet)	
	Jamal Righi	(Alcoa)	
	Wolfgang Schneider	(VAW)	
	Per Arne Tondel	(Elkem)	
	Diane Zekind	(Hayes Lemmerz)	

PROJECT STATEMENT

Objectives

- Model thermodynamic phase equilibria for alternate alloy systems, such as aluminum-silicon-magnesium, aluminum-magnesium, and aluminum-copper.
- Model solidification behavior and determine key characteristics.
- Use the information on solidification behavior to tailor and develop alloys that are better suited for SSM processing.

Strategy

- Use Thermocalc and Dictra thermodynamic modeling packages together with an aluminum alloy database to determine phase equilibria for SSM alloys.
- Identify key characteristics of the alloy that can be determined from model.
- Identify desired characteristics of an ideal SSM alloy.

- Use thermodynamic model to develop alloy with ideal characteristics.
- Carry out tests on new alloys to provide feedback and validate results.

ACHIEVEMENTS THIS QUARTER

- Solid fraction and solid fraction variation as a function of processing temperature has been simulated for 500 series of alloys over the composition range of 1~10wt% magnesium and 0.05~3wt% of silicon.
- A region in the composition range where there is a peak of solid fraction variation has been identified. The peak value increases with increasing silicon concentration and decreasing magnesium concentration.
- Samples have been prepared for measurements of composition distribution using electron microprobe analysis in order to validate the thermodynamic simulations.

CHANGES IN PROJECT STATEMENTS

None

WORK PLANNED FOR NEXT QUARTER

- Carry out industrial and/or experimental validation of new SSM alloys.

OPERATIONAL SCHEDULE

	Apr-01	May-01	Jun-01	Jul-01	Aug-01
Identify Alternate Al-Mg Alloys					
Conduct Validation Studies					

Development of Alternate SSM Alloys

Q. Han and S. Viswanathan
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, TN 37831-6083

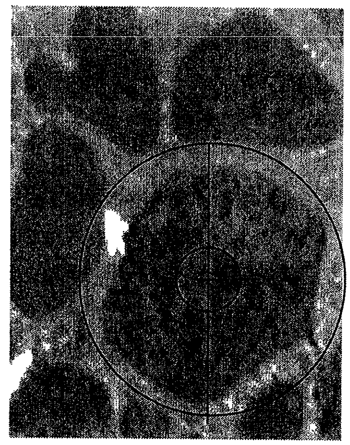
Review Meeting
Worcester, May 15, 2001

Work Since The Last Review Meeting Addresses Two Issues

- Validation of Thermodynamic Predictions in SSM 357 Alloy
  - Composition distribution in the primary phase and in the eutectic regions.
- Simulations of Al-Mg (500 series) alloys.
  - Alloys are Al - (1~10)Mg - (1~3)Si - 0.5Fe - 0.15Cu - 0.35Mn - 0.15Zn - 0.25Ti
  - **Solid fractions** and **solid fraction variations** as a function of processing temperatures.

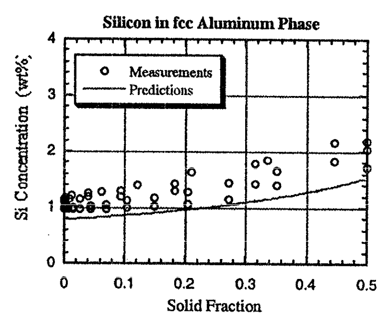
Thermodynamic Predictions of Solute Distribution in 357 Alloy Have Been Validated

- JEOL 773 Electron microprobe was used to measure solute distribution across grains in SSM Billet.
- The distance across a grain was converted into solid fraction, a non-dimensional parameter. The solid fraction at the edge of the grain was assumed to be 0.5, since eutectic occurs at 0.5 solid fraction.
- The measurements were compared with calculations made assuming Scheil behavior.



Prediction of Silicon Distribution Agrees Well with Measurements

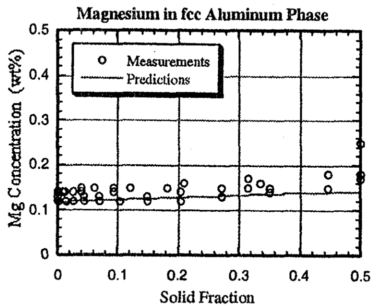
- Measurements of silicon concentration are higher than predictions.
  - It is unlikely that grains were sectioned at the center.
- The minimum values of the measurement fit the predictions.
- Back diffusion is not included in the model.
- The exact ingot composition is not known.



Alloy composition (wt%):
Si 7.0, Mg 0.55, Fe 0.1, Ti 0.06
Cu 0.008, Mn 0.0005, Zn 0.001

Prediction of Magnesium Distribution Agrees Well with Measurements

- The minimum value of the measured Mg concentration matches the prediction.
- Magnesium concentration in the grain is only 1/3 of the bulk concentration.

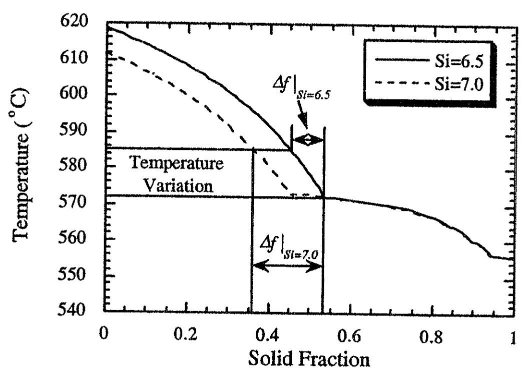


Alloy composition (wt%):
Si 7.0, Mg 0.55, Fe 0.1, Ti 0.06
Cu 0.008, Mn 0.0005, Zn 0.001

Simulation of Al-Mg (500 Series) Alloys Has Been Completed

- Solid fraction versus temperature curves for alloys in the composition range of 1-10wt% Mg and 1-3wt% Si have been determined.
- Solid fraction variation for Al-Mg (500 series) alloys as a function of processing temperature has been calculated.
- Al-Mg alloys appear to have good potential for semi-solid processing.
- It appears that Al-Mg alloys can be processed at solid fractions greater than 0.5.

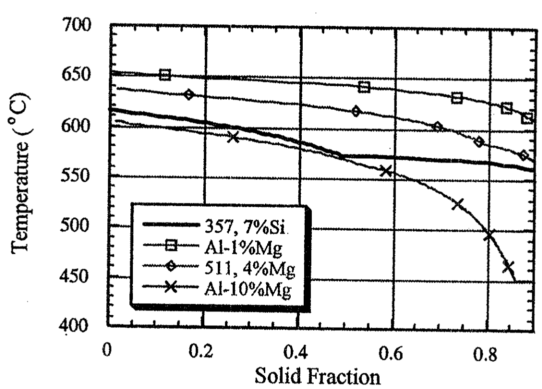
Both the Solid Fraction and the Solid Fraction Variation with Temperature Affect SSM Processing



Note

A small solid fraction variation indicates a large processing window at that temperature.

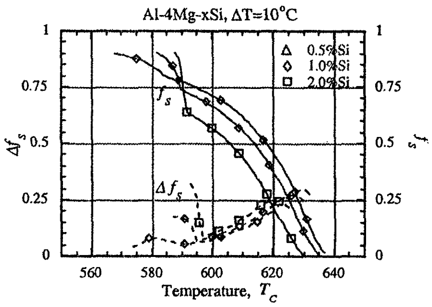
The Solid Fraction Curve of Al-Mg Alloys Is Very Different Compared to Al-Si Alloy



6

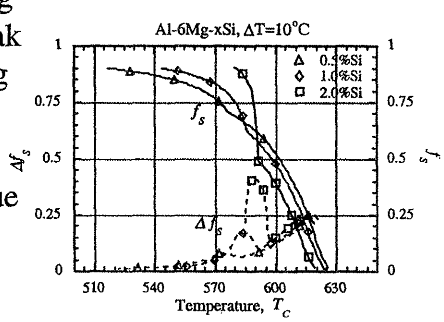
Al-4%Mg-(0.5~2)%Si Alloys Appear to Have Potential for SSM Processing

- When the silicon concentration is low, the solid fraction variation decreases with decreasing processing temperatures.
- When the silicon concentration is higher than 1wt%, there is a sharp increase in solid fraction variation when the processing temperature is below 600°C.
- The alloys may be processed at a solid fraction greater than 0.5.



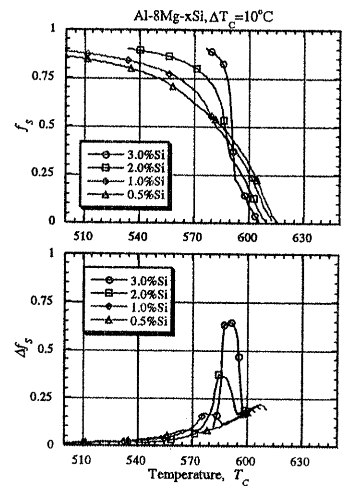
Al-6%Mg-(0.5~2)%Si Alloys Have Potential at High Solid Fraction or Low Si

- A peak in solid fraction variation occurs at a processing temperature of 590°C. The peak value increases with increasing silicon concentration.
- The solid fraction corresponding to the peak value is about 0.6.
- The smallest solid fraction variation is obtained at high solid fraction (>0.75).



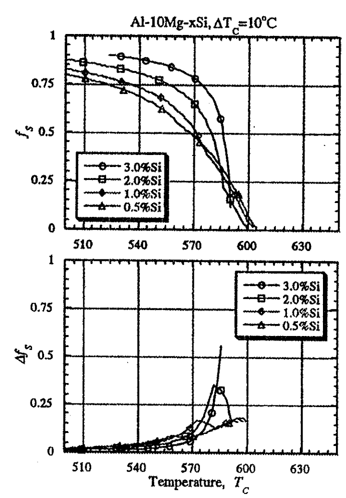
Al-8%Mg-(0.5~3)%Si Alloys Can Be Processed at $f_s >= 0.6$

- The peak in solid fraction variation, which occurs at 590°C, increases with silicon concentration.
- When the solid fraction is higher than 0.5, the solid fraction variation decreases with decreasing processing temperatures.



Al-10%Mg-(0.5~3)%Si Alloys Can Be Processed at $f_s >= 0.5$

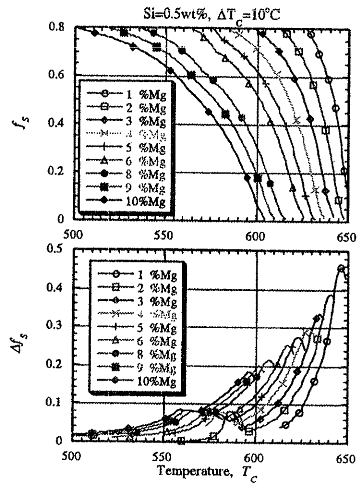
- For Si%<1, the solid fraction variation decreases with processing temperature.
- For Si%>2, a peak in solid fraction variation occurs at a solid fraction less than 0.5.
- The alloys may be processed at high solid fraction with low solid fraction variation.



8

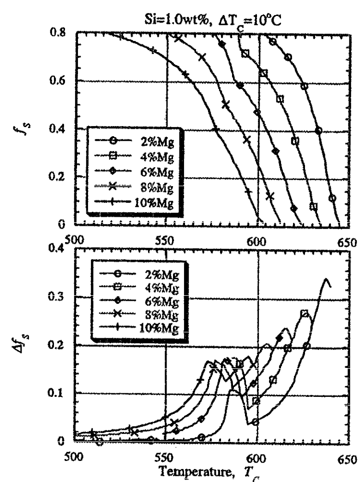
The Solid Fraction Variation Decreases with Decreasing Processing Temperature When Si%=0.05

- Solid fraction variation decreases with decreasing processing temperatures, indicating that the alloys should be processed at higher solid fraction.
- The peak shifts to lower processing temperatures with increasing magnesium contents.



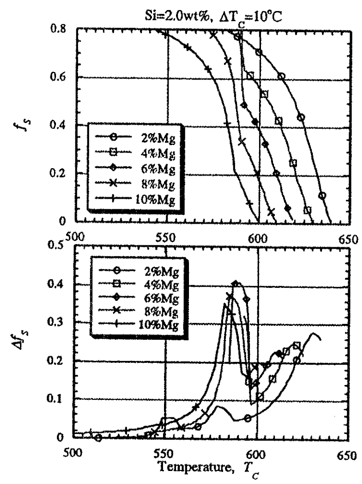
Increased Peak Values of Solid Fraction Variation Occur When Si=1wt%

- With increasing magnesium concentration, the peak is shifted to lower processing temperatures.
- To avoid high solid fraction variation in 500 series alloys, the magnesium concentration should be either lower or higher than the region of the peak, when the silicon concentration is about 1 wt%.

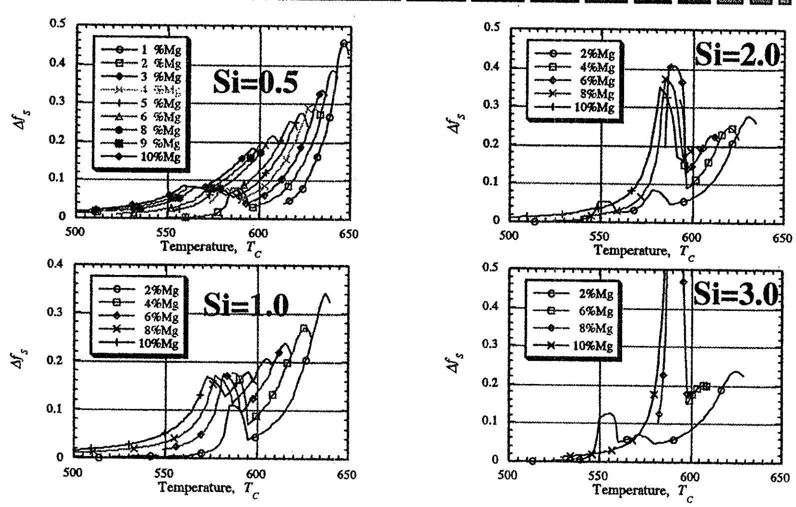


High Peak Values Occur around 590°C When Si=2 wt%

- The peak in solid fraction variation is as high as 0.3~0.4.
- Alloys of low Mg (<4%) or high Mg content (10%) can still be processed at low solid fraction variation.
- When Mg is 6-8%, the alloy must be processed at low solid fraction (<0.4) to avoid high solid fraction variation.



The Peak in Solid Fraction Variation Increases With Increasing Silicon Concentration



Al-Mg Alloys Can be Processed at Low Si or High Solid Fraction

- Solid fraction and solid fraction variation as a function of processing temperature over the whole composition range of Al-Mg alloys have been computed.
- The alloys can be processed at high solid fractions. Alloys with Si < 0.5% can be processed at a wide range of solid fractions.
- A peak in solid fraction variation has been identified for each composition when Si > 0.5%. The peak occurs around 560~590°C depending on the alloy composition.
- The processing temperature of 500 series alloys should be determined such that the peak in solid fraction variation is avoided.

Future Work

- Validation of low silicon 357 alloys.
  - Test of low silicon (5.5~6.5%) 357 alloys for less liquid drip and improved process consistency.
- Dilatometry tests of 357 SSM alloys and squeeze cast alloys.
  - Compare dynamic behavior of these two alloys during T4 and T5.
  - Thermodynamic and kinetic modeling if necessary.
- Suggestions for SSM 500 series alloys.